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Indian Standard

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भाग 1 हाथ से नमूने लेना

(दूसरा पुनरीक्षण)

Methods of Sampling of Petroleum and its Products

Part 1 Manual Sampling

(Second Revision)

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Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Samples of petroleum and petroleum products are examined by various methods of test for determination of physical and chemical characteristics. The test results are often used for custody transfer and pricing determinations. It is accordingly necessary that the samples be representative of the petroleum or petroleum products in question. The precautions required to ensure the representative character of the samples are numerous and depend upon the type of material being sampled the tank, carrier, container or line from which the sample is being obtained, the type and the cleanliness of the sample container, and the sampling procedure that is to be used. A summary of the sampling procedures and their applications may be given as follows:

Sampling Procedure	*Type of Container*	*Application*
Pre-cooled bottle sampling	Storage tanks, ship and barge tanks, tank cars, tank trucks	Liquids of more than 110 kPa and not more than 179 kPa RVP
Cooler tap sampling	Storage tanks with taps	do
Bottle sampling	Storage tanks, ship and barge tanks, tank cars, tank trucks	Liquids of more than 13.8 kPa and not more than 110 kPa RVP
Tap sampling	Storage tanks with taps	do
In-line sampling	Pipes or lines	Liquids of 110 kPa or less RVP
Bottle sampling	Storage tanks, ship and barge	Liquids of 13.8 kPa or less RVP
Tap sampling	Storage tanks with taps	do
Dipper sampling	Free or open-discharge streams	do
Tube sampling	Drums, barrels and cans	do
Thief sampling	Tank cars, storage tanks	Bottom or thief sampling of liquids of 13.8 kPa RVP or less
Dipper sampling	Free or open-discharge streams, open tanks or kettles with open heads; tank cars and tank trucks; drums	Liquids or semi-liquids 13.8 kPa RVP or less
Automatic sampling, thief sampling; bottle sampling; tap sampling	Storage tanks, ship and barge tanks, tank cars, tank trucks, and pipelines	Crude petroleum
Bottle sampling	Storage tanks, ship and barge tanks	Industrial aromatic hydrocarbons
Boring sampling	Barrels, cases, bags, cakes	Waxes, solid bitumens, and other soft solids
Grab sampling	Freight cars, conveyors bags, barrels, boxes	Petroleum coke; lumpy solids
Grease sampling	Kettles drums, cans tubes	Greases, soft waxes, asphalts
	Storage tanks, tanks, cars, lines, packages	Asphaltic materials
	Storage tanks, tank cars, lines, packages	Emulsified asphalts
Extended-tube sampling thief sampling	Ship and barge trucks, storage tanks	Bottom water composition

(*Continued to third cover*)

Indian Standard

METHODS OF SAMPLING OF PETROLEUM AND ITS PRODUCTS

PART 1 MANUAL SAMPLING

(Second Revision)

1 SCOPE

1.1 This standard (Part 1) covers procedures and equipment for manually obtaining representative samples of liquid petroleum products, intermediate products and crude oils. This standard also covers sampling of semi liquid petroleum products.

1.2 This standard does not cover sampling of electrical insulating oils and fluid power hydraulic fluids.

1.3 Different degrees of uniformity or the presence of sediment and water are shown to present manual sampling difficulties that may affect the representative nature of final samples.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of regulatory limitations and any other restrictions for this purpose.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute the provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
1448	Methods of test for petroleum and its products
(Part 18) : 2020	Distillation of petroleum products (*third revision*)
(Part 39) : 2012/ ISO 3007 : 1999	Determination of vapour pressure — Reid method (*second revision*)
(Part 148) : 2019/ ISO 6297 : 1997	Petroleum products — Aviation and distillate fuels — Determination of electrical conductivity (*first revision*)

3 TERMINOLOGY

3.1 Samples

3.1.1 *Uniform Petroleum Product* — One in which spot samples from top, upper, middle, lower, and outlet agree within the precision of the laboratory tests. Similarly, in pipeline transfers, spot samples taken at 1, 20, 50 and 80 percent of the total volume agree within the precision of the laboratory tests.

3.1.2 *Official Sample* — A sample taken at the point of custody transfer and used for the custody transfer laboratory determination.

3.1.3 *Representative Sample* — A portion extracted from the total volume that contains the constituents in the same proportion that are present in that total volume.

3.1.4 *All Levels Sample* — A sample obtained by submerging a stoppered beaker or bottle to a point as near as possible to the draw-off level, then opening the sampler and raising it at a rate such that it is about three-fourths full (maximum 85 percent) as it emerges from the liquid. An all-levels example is not necessarily a representative sample because the tank volume may not be proportional to the depth and because the operator may not be able to raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion.

NOTE — The tube sampling procedure may be (*see* **7.6**) used to obtain on all levels samples from a barrel or drum.

3.1.5 *Running Sample* — A sample obtained by lowering an un stoppered beaker or bottle from the top of the oil to the level of the bottom of the outlet connection or swing line, and returning it to the top of the oil at a uniform rate of speed such that the beaker or bottle is about three-fourths full when withdrawn from the oil. A running sample is not necessarily a representative sample because the tank volume may not be proportional to the depth and because the operator may not be able to raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion.

3.1.6 *Spot Sample* — A sample taken at a specific location in tank or from a flowing stream in a pipe at a specific time during a pumping operation.

3.1.7 *Top Sample* — A spot sample obtained 150 mm below the top surface of the liquid (*see* Fig. 1).

3.1.8 *Upper Sample* — A spot sample taken at the middle of the upper one-third of the tank contents (a distance of one-sixth of the liquid depth below the liquid's surface) as shown in Fig. 1.

NOTE — The outlet sample location shown applies only to tanks with side outlets. It does not apply when the outlet comes from the floor of the tank or turns down into a sump.

3.1.9 *Middle Sample* — A spot sample obtained from the middle of the tank contents (a distance of one half of the depth of liquid below the liquid's surface) (*see* Fig. 1).

3.1.10 *Lower Sample* — A spot sample obtained from the middle of the lower one-third of the tank contents (a distance of five-sixth of the depth of liquid below the liquid's surface) (*see* Fig. 1).

3.1.11 *Clearance Sample* — A spot sample taken with the inlet opening of the sampling apparatus 100 mm below the bottom of the tank outlet.

3.1.12 *Bottom Sample* — A spot sample obtained from the material at the bottom surface of the tank, container or line at its lowest point.

3.1.13 *Bottom Water Sample* — A spot sample of free water taken from beneath the petroleum contained in a ship or barge compartment or storage tank.

3.1.14 *Drain Sample* — A sample obtained from the water draw-off valve on a storage tank. Occasionally, a drain sample may be the same as a bottom sample, as in the case of a tank car.

3.1.15 *Outlet Sample* — A spot sample taken at the level of the bottom of the tank outlet (either fixed or swing pipe) but not higher than one meter above the bottom of the tank (*see* Fig. 1).

3.1.16 *Automatic Sampler* — A sampler used to retrieve a representative sample from the liquid flowing in a pipe. The automatic sampler generally consists of a probe, an extracting mechanism, an associated controller, and a sample receiver.

3.1.17 *Single Tank Composite Sample* — A blend created from the upper, middle, and lower samples from a single tank. For a tank of uniform cross-section, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. For a horizontal cylindrical tank, the blend consists of the three samples in the proportions shown in Table 1.

3.1.18 *Multiple Tank Composite Samples (Ships, Barges, etc.)* — A mixture of individual samples from the several compartments each of which contains the same grade of petroleum material. The mixture is blended in proportion to the volume of material in each compartment.

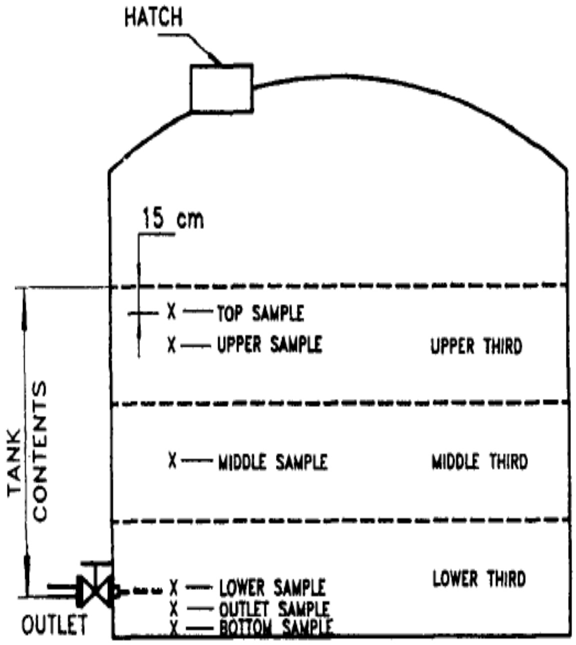


FIG.1 SAMPLING DEPTHS

Table 1 Sampling Instructions for Horizontal Cylindrical Tests

(*Clause* 3.1.17)

Sl No.	Liquid Depth, Percent of Diameter	Sampling Level, Percent of Diameter Above			Composite Sample, Proportionate Parts of		
		Upper	Middle	Lower	Upper	Middle	Lower
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	100	80	50	20	3	4	3
ii)	90	75	50	20	3	4	3
iii)	80	70	50	20	2	5	3
iv)	70		50	20		6	4
v)	60		50	20		5	5
vi)	50		40	20		4	6
vii)	40			20			10
viii)	30			15			10
ix)	20			10			10
x)	10			5			10

3.1.19 *Composite Spot Sample* — A blend of spot samples mixed in proportion to the volumes of materials from which the spot samples were obtained. Some tests may also be made on the spot samples before blending and the results averaged. Spot samples from crude oil tanks are collected as follows.

3.1.19.1 *Three-way* — On tanks larger than 1 000 barrel capacity that contain in excess 4.5 m of oil, equal volume samples should be taken at the upper, middle and lower or outlet connection of the merchantable oil, in the order named. On tanks of 1 000 barrel capacity and less, this method may also be used.

3.1.19.2 *Two-way* — On tanks smaller than 1 000 barrel capacity that contain in excess of 3.0 m and up to 4.5 m of oil, equal-volume samples should be taken at the upper and lower, or outlet, connection of the merchantable oil, in the order named. On tanks of 159 m^3 (1 000 barrel) capacity and under, this method may also be used.

3.1.20 *Middle Spot Sample* — On tanks larger than 1 000 barrel capacity containing 3.0 m or less of crude oil, one spot sample should be taken near the centre of the vertical column of oil as possible and outlet connection.

3.1.21 *Dipper Sample* — A sample obtained by placing a dipper or other collecting vessel into the path of a free flowing stream, so as to collect a definite volume from the full cross-section of the stream, at regular time intervals for a constant rate of flow, or at time intervals varied in proportion to the rate of flow.

3.1.22 *Tube or Thief Sample* — A sample obtained with a sampling tube or special thief, either as a core sample or spot sample from a specific point in the tank or container.

3.1.23 *Tank Side Sample* — A spot sample taken from a sample connection in the side of a tank.

3.1.24 *Surface Sample (Skim Sample)* — A spot sample skimmed from the surface of a liquid in a tank.

3.1.25 *Flow Proportional Sample* — A sample taken from a pipe such that the rate of sampling is proportional throughout the sampling period to the flow rate of the fluid in the pipe.

3.1.26 *Entrained Water* — Water suspended in the oil. Entrained water includes emulsions but does not include dissolved water.

3.1.27 *Free Water* — Water that exists as a separate phase and typically lies beneath the oil.

3.1.28 *Emulsion* — An oil/water mixture that does not readily separate.

3.1.29 *Sample Receiver (Receptacle)* — Container, in which all sample bites are collected during sampling operation. A receiver may be fixed or portable.

3.1.30 *Boring Sample* — A sample obtained by collecting the chips made by boring holes with a ship auger from top to bottom of the material contained in a barrel, case, bag or cake.

3.1.31 *Zone Sample* — Sample taken as that part of the liquid column that is trapped within the whole height of a sampling device when it is sealed at a single spot location within a tank after having been fully flushed as it was lowered to that position.

3.1.32 *Nozzle Sample* — Nozzle sample is one obtained from fuel dispensing nozzle of fuel station/retail outlet/petrol pump which dispenses fuel from the underground storage tank. The nozzle sampling is applicable for sampling of product from a fuel station/retail outlet/petrol pump or wherever fuel dispensing system is available.

3.1.33 *Homogeneity* — Homogeneity is the quality of uniformity of a material. If irregularities are distributed evenly in a mixture of material, the material is homogeneous. Product shall be termed as homogeneous if density variation between UML or TMB is not more than ± 3.0 kg/m^3 between any two layers.

3.2 Vertical Cylindrical Tanks

3.2.1 *Spot Samples*

Lower the sampling device until its opening is at the required depth, open it in the appropriate manner and maintain it at the required level until it is filled. Retrieve it and either decant a small portion back to the tank to create an ullage space before sealing the sample, or carefully transfer the complete sample to a secondary sample receiver.

In hot climate conditions or whenever there is a significant difference in temperature between the sampler and the product being sampled, the sampler should be conditioned to the temperature of the tank contents by gently raising and lowering it through approximately ±300 mm for 1 min to 2 min before operating the opening mechanism.

When sampling at different levels, take the samples in sequence from top to bottom, in order to avoid disturbance at a lower level.

In the case of a zone sampler (with essentially full bore top and bottom valves that allow the tank contents to flush through the sampler as it is lowered), lower the sampler in a controlled manner until it is at the required depth. Close the valve(s) as soon as the lowering operation ceases, and retrieve the sample immediately. Carefully transfer the complete sample to a secondary sample receiver.

Where the design of the zone sampler does not permit full flushing during lowering, it is recommended that the sampler be raised and lowered two or three times after reaching the spot sampling location, before closing the valve(s). The raising and lowering shall be through a distance of at least the height of the sampler.

In the case of a top sample, lower the open sampler/container carefully until its neck is just above the surface of the liquid, and then allow the sampler to fall sharply 150 mm below the surface. When the sampler is full, as indicated by the cessation of air bubbles, withdraw it and proceed as for regular spot samples.

3.2.2 *Composite Samples*

A composite sample may be prepared from representative subsamples of spot samples obtained from within a single tank (for example, by combining subsamples from the upper, middle and lower spot samples), or may be prepared by combining subsamples representative of individual tanks to provide a composite for a larger oil quantity (for example, several ship or barge tanks of the same product). Composite samples shall include all the material collected in the primary sampling device without subdivision. The volume collected in the primary sampling device shall be chosen to allow the entire contents of the device to be added to the volume of the other subsamples in the transport container. Compositing of subsamples smaller than the entire contents of a subsample shall only be performed in a laboratory with facilities to assure adequate mixing and measurement of the subsamples.

To prepare either type of composite sample, transfer subsamples of representative individual samples into a composite-sample container, and mix them together gently. The subsamples shall be volume weighted in proportion to the quantity that each represents.

When the subsamples that are to be combined originate from a tank of non-uniform cross-sectional area (or from multiple tanks), the compositing operation shall require careful calculation and measurement of the subsamples to maintain the representative nature of the sample. These operations shall, if practical, be conducted under controlled laboratory conditions.

NOTE — Evaporation of light ends, and adhesion of water/sediment to the wall of the original sampler, may influence the representative nature of composite sample(s).

Do not prepare composite samples for testing unless they are specifically requested, and agreed by the interested parties. As an alternative to physical compositing, individual spot samples shall be tested and a mean value calculated from the individual test results in proportion to the bulk represented by each sample.

3.2.3 *Bottom Sampling*

Lower the bottom sampler until it rests in an upright position on the bottom of the tank so that the valve opens and the sampler fills. After withdrawing the sampler, examine it closely for leaks. If any are detected, discard the sample, clean the bottom sampler and re-sample. If necessary, transfer the contents into a secondary sample container, taking care that all the sample is properly transferred, including any water or solids that may adhere to the inner walls of the primary bottom sampler.

3.2.4 *Interface Sampling*

Lower the sampler with the valves open to permit the liquid to flush through the device. At the level desired, close the valve(s) and withdraw the sampler from the liquid.

If a transparent tube is used, any existing interface can be detected visually through the wall of the sampling tube and its position within the tank determined by measurement on the graduated sampling tape. Check that the valves have properly closed; otherwise re-sample.

NOTE — The sample may be retained for testing.

3.2.5 *Tap (Tank-side) Sampling*

This is not a preferred method for custody transfer or yield inventory purposes and therefore shall be applied only if no other method of sampling is possible.

The sampling point valves shall be a minimum of 12.5 mm in diameter and shall be fitted to the side of the tank at regular intervals, by connections extending at least 150 mm into the tank, except on floating-roof tanks, in which this is impossible. The lower connection shall be level with the bottom of the suction pipe.

Before a sample is taken, flush the tap or valve connection with the product to be sampled, after which draw off a sample into a container or receiver.

CAUTION — Open the taps with care when sampling under pressure. Make no attempt to clear a blocked connection by rodding through an opened valve.

If the contents of a tank fail to reach the upper or middle sample connections on a tank equipped with three connections, take the sample for the tank as follows:

a) If the level of the contents is nearer the upper sample connection than the middle one, take two-thirds of the sample from the middle connection and one-third from the lower one.
b) If the level is nearer the middle connection than the upper, take one-half of the sample from the middle connection and one-half from the lower one. If the level of the contents is below the middle sample connection, take all the sample from the lower connection.

3.2.6 *All-level Sampling*

All-level samplers can be of the ‘top-down’ or ‘bottom-up’ type. As the sample receiver is filled while travelling in a single direction through the tank’s contents, different procedures are required for ‘top-down’ and ‘bottom-up’ techniques.

Follow the manufacturer’s instructions when using proprietary devices.

To obtain a (bottom-up) all-level sample with a bottle fitted to a weighted sampling cage (or with a weighted sampling can), proceed as follows. Stopper the bottle or can, and lower it to the tank bottom (avoiding any bottom-lying free water). Jerk the cord to remove the cork, and raise the sampler back to the surface at a uniform speed without any pause or hesitation. The hauling speed should be chosen so that the bottle or can is about 80 percent full, but not more than 90 percent full, when withdrawn from the liquid. Cap or stopper the bottle immediately, or carefully transfer the complete sample from the weighted can to a secondary transportation receiver.

If a fixed volume all-level sampler is less than 90 percent full when retrieved from the liquid, it may be assumed that oil was flowing into it from all depths during its passage through the tank’s contents. If the sampler is more than 90 percent full when retrieved from the liquid, the sample may not be representative and should be discarded before re-taking the sample using a faster raising speed.

4 SUMMARY OF PRACTICE

4.1 This practice provides procedures for manually obtaining samples of petroleum and petroleum products of a liquid, semi solids or solid state samples from tanks, pipelines, drums, barrels, cans, tubes. It addresses in details, the various factors which need to be considered in obtaining a representative sample.

4.1.1 If the tank contents are not homogeneous from top to bottom of the tank or if the conditions in **4.1.2** are not met, automatic sampling is recommended.

4.1.2 Tank samples will be representative if the tank contents are homogeneous from top to bottom. This is rarely the case in actual practice. However, tank samples are acceptable if all of the following conditions prevail:

a) Tank contains a heavy component (such as water) that clearly separates from the main component;
b) Tank is equipped with either a swing suction or a weir on the outlet that prevents any shipment of the heavy component; and
c) Tank samples are taken so that none of the heavy component is included.

4.1.3 *See* **6** for additional precautions and instructions.

5 APPARATUS

5.1 Sample Containers

Sample containers come in various shapes, sizes and materials. Select the proper container based on the product to be sampled. To select the right container for a given application, knowledge of the material to be sampled is required to ensure that there will be no interaction between the sampled material and the container which would affect the integrity of the sample. Type of mixing required to remix the contents before transferring the sample from the container is additional consideration required while choosing the container type.

In general clear or brown glass bottles, or cans may be used. The clear bottle is advantageous because it may be examined visually for cleanliness and also allows visual inspection of the sample for free water or solid impurities. The brown glass bottle affords some protection from light.

Cans made up of stainless steel with welded seams and aluminium bottle containers are suitable for many sampling operations.

The following are general design considerations for choosing the sample container:

a) The bottom of the container should be sloped continuously downwards to the outlet to ensure complete liquid withdrawal.
b) There should be no internal pockets or dead spots.
c) Internal surfaces should be designed to minimize corrosion, encrustation, and water/sediment clingage.
d) There should be an inspection cover/closure of sufficient size to facilitate filling, inspection, and cleaning.
e) The container should be designed to allow the preparation of a homogeneous mixture of the sample while preventing the loss of any constituents which affect the representativeness of the sample and the accuracy of the analytical tests.
f) The container should be designed to allow the transfer of samples from the container to the analytical apparatus while maintaining their representative nature.

5.1.1 *Plastic Bottles*

Plastic bottles made of suitable material (un-pigmented linear polyethylene) may be used for handling and storage of diesel oil, fuel oil and lubricating oils. Bottles of this type should not be used for gasoline, aviation jet fuel, kerosene, crude oil, white spirit or other petroleum products unless testing indicates there is no problem with solubility, contamination or loss of light components. In no circumstances shall nonlinear conventional polyethylene containers be used to store samples of liquid hydrocarbon. Used engine oil samples that may have been subjected to fuel dilution shall not be stored in plastic containers. Plastic bottles have an advantage. They will not shatter like glass or corrode like metal containers. They are generally used only once and discarded so that re-cleaning and recovery procedures are not required.

5.1.2 *Sample Containers for Aviation Fuels*

A number of aviation fuel properties are affected by trace levels of polar or other compounds. Therefore, the sample containers used should not add or absorb any material.

5.1.2.1 Epoxy coated cans are generally considered satisfactory for aviation gasoline.

5.1.2.2 Amber coloured glass bottles or glass bottles covered with opaque material such as aluminium foil are used as sample containers for aviation fuels.

5.1.2.3 Because no specification is known to describe a satisfactory epoxy-coating, below listed procedures is an approval procedure, which can be used to identify a satisfactory coating. For initial qualification of new container sources, coated cans should be examined closely to assure that the coating covers all inside surfaces.

The sample containers used for Jet A-1 may be acceptable, if they comply to the following test:

a) The container to be filled with Jet A-1 and stored for at least one month at room temperature. During this period the sample should be shaken strongly at least once a week. At the end of storage, the sample to be tested for electrical conductivity and water separation. The final electrical conductivity should not be higher by more than 2 pS/m from the initial value. The water separation rating should decrease by no more than three MSEP units.
b) Supplemental testing is necessary if the fuel normally contains additives such as conductivity improvers which may be desorbed. In that case a large additive-containing sample which has been stored for a month or longer to equilibrate additive content should be used as the test fuel. Such fuel should have a conductivity above 50 pS/m, if the additive is conductivity improver additive; and the MSEP value should also be determined. After similar storage for at least one month, the final electrical conductivity should not change more than the repeatability limits of test method IS 1448 (Part 148). The final MSEP rating should be within the repeatability limits for the initially obtained value.

5.1.2.4 The summary of recommendation for various containers of aviation fuel is given in Annex B.

5.2 Container Closure

Cork stoppers, or screw caps of plastic or metal may be used for glass bottles. Corks must be of good quality, clean, and free of holes and loose bits of cork. Situations in which cork should not be used include liquids in which loss of light ends may affect any test result and liquids that are hygroscopic or have a low water content specification.

Never use rubber stoppers. Prevent the sample from contacting the cork by wrapping tin or aluminium foil around the cork before forcing it into the bottle. Screw caps made of a material that will not deteriorate or contaminate the sample are to be used for glass bottles. Screw caps should provide a vapour tight seal.

Glass stoppers must be a perfect fit. Cans and plastic bottles should be closed with screw caps made of the same material as the container. Screw caps must be protected by a disk faced with tin or aluminum foil, or other material that will not affect petroleum or petroleum products.

5.3 Cleaning Procedure

All sample containers must be absolutely clean and free of water, dirt, lint, washing compounds, naphtha, or other solvents, soldering fluxes or acids, corrosion, rust, or oil. Before using a container rinse it with standard solvent or other naphtha of similar volatility (It may be necessary to use sludge solvent to remove all traces of sediment and sludge from the container previously used). Following the solvent wash, the container should be washed with a strong soap solution, rinsed thoroughly with tap water, and given a final rinse using distilled water. Dry the container either by passing a current of clean, warm air through the container or by placing it in a hot dust-free cabinet at 40 °C or higher. When dry, stopper or cap the container immediately. In the ordinary sampling of crude petroleum, washing with soap and rinsing with water may be eliminated. Finally, container shall be thoroughly rinsed with product.

5.4 Sampling Cage

This shall be a metal or plastic holder or cage, suitably constructed to hold the appropriate container. The combined apparatus shall be of such a weight as to sink readily in the material to be sampled, and provision shall be made to fill the container at any desired level (*see* Fig. 2). Bottles of special dimensions are required to fit a sampling cage. The use of sampling cage is generally preferred to that of a weighted sampling beaker for volatile products since loss of light ends is likely to occur when transferring the sample from a weighted sampling beaker to another container.

5.5 Sampling Apparatus

Described in detail under each of the specific sampling procedures. Sampling apparatus shall be clean, dry and free of all substances that might contaminate the material.

6 PRECAUTIONS AND INSTRUCTIONS FOR SPECIFIC PRODUCTS

6.1 Sampling certain products requires a due amount of caution for their handling. *See* Annex A for precautionary statements regarding these products.

6.2 Crude Petroleum and Heavy Fuels

6.2.1 Usually are non-homogeneous. Automatic samplers are recommended for sediment and water (S&W) and density measurement. Tank samples may not be representative because of the following reasons.

6.2.1.1 The concentration of entrained water is higher near the bottom. The running sample or the composite of the upper, middle and lower sample may not represent the concentration of entrained water.

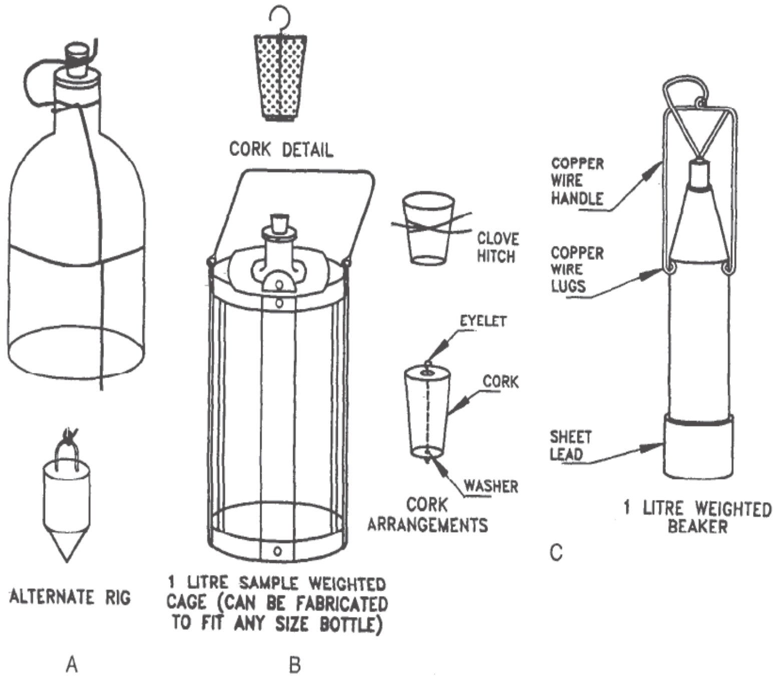


FIG. 2 ASSEMBLIES FOR BOTTLE SAMPLING

6.2.1.2 The interface between oil and free water is difficult to measure, especially, in the presence of emulsion, layers, or sludge.

6.2.1.3 Determining the volume of free water is difficult because the free water level varies across the tank bottom surface. The bottom is often covered by pools of free water or water emulsion impounded by layers of sludge or wax.

6.2.1.4 In case of high pour and high viscous crudes exercise great care with respect to heating and homogenizing to ensure that sample remains representative during all handling stages from primary container to test apparatus.

6.3 Gasoline and Distillate Products

Usually are homogeneous but they are often shipped from tanks that have clearly separated water on the bottom. Tank sampling is acceptable under conditions covered in Foreword.

6.4 When using tank samples, the S&W deduction is usually the sum of the free water volume (usually determined from a paste cut) and the entrained water volume determined from the S&W analysis of the tank sample. The difficulty of determining the free water volume limits the accuracy of the S&W deduction.

6.4.1 Automatic samples are recommended. However, by mutual agreement, shore tanks, ship(s) compartment or manual line samples may be used.

6.5 Aviation Fuels

6.5.1 Sampling equipment fabricated from copper and its alloys shall not be used for sampling aviation fuels.

6.5.2 Before sampling, the sampler and the container shall be flushed and rinsed thoroughly at least three times with the product to be sampled and allowed to be drained before use.

6.5.3 Certain container types are found suitable for some test and not for others. Some containers are adequate for immediate use and not for storage. A summary of recommended containers is provided in Annex B.

6.5.4 Electrical conductivity of jet fuels is known to change during storage. Results obtained from shipped samples may not be a reliable indicator for the fuel's actual conductivity level. Therefore, conductivity test should be carried out directly on the fuel in tankage or by drawing a sample for immediate testing.

6.6 Sample Handling and Dividing of Crude Petroleum and Non-Uniform Products

6.6.1 The transfer of crude oil samples from the sample receiver to the laboratory glassware in which they will be analyzed requires special care to maintain their representative nature. The number of transfers should be minimized. Mechanical means of mixing and transferring the samples are recommended. An external circulating system provided with pump and in-line mixing element that couples directly to the sample container is one method. Mixing time and flow rates are critical in such systems. A mixer that is inserted in the sampler container is another method. Whatever handling, dividing, and mixing system is chosen, its performance should be verified by introducing and transferring samples of known water concentration.

6.6.2 In mixing and transferring, representative samples; the variation from known water concentration should not exceed ± 0.05 percent when the known water concentration is 1 percent or less and ± 5 percent of the known water concentration when it is over 1 percent.

6.7 Marine Custody Transfer

By mutual agreement, samples can be taken from shore tanks, from ship(s) tanks, or from pipelines. Pipelines samples may be taken automatically or manually. Properly taken automatic pipeline samples are the most representative. Manual pipeline samples are less representative than automatic pipeline samples. Manual pipeline sampling is described in **7.4**. Tank samples will usually not be representative unless the tank is completely homogeneous and contains no free water.

6.7.1 *Stationary or Shore Tanks*

6.7.1.1 Crude petroleum tanks may be sampled in the following ways by mutual agreement, composite spot, middle spot, all levels, running samples or by sample cocks. Additional samples may be taken as necessary.

NOTE — Where emulsions are in relatively higher concentration in the bottom portions of the tank, the lower sample would not be considered representative of this lower third. Automatic line sampling is recommended in such cases. If this is not possible, an outlet sample or bottom sample should be required instead of the lower sample. In addition, a bottom thieving should be made for both opening and closing gauges so that any change in the S&W level at the bottom of the tank may be observed and noted.

6.7.1.2 Where tank samples must be used for crude oil fiscalization and the tanks do not have swing suction lines or weirs, it is recommended that upper, middle, and outlet samples be taken. These samples should be tested and reported separately. The S&W deduction should be the average of the three values. Other analyses should also be averaged.

6.7.2 *Ship or Barge Transfers*

Samples of ship cargoes of crude petroleum may be taken by the following methods by mutual agreement.

6.7.2.1 From the shore tanks before loading and both before and after discharging as previously described.

6.7.2.2 From the pipeline during discharging or loading, pipeline samples may be taken either manually or with an automatic sampler. If the pipeline requires displacement or flushing, care must be taken that the pipeline sample includes the entire cargo and none of the displacement. Separate samples may be required to cover the effect of the line displacement on the prior or following transfer.

6.7.2.3 From the ship(s) tanks after loading or before discharging, an all-levels sample or a running sample shall be obtained from each compartment of the ship(s) cargo tanks. Except for the following:

a) Except where specifically exempted, when loading a ship, the shore tank sample or the pipeline sample taken from the loading line shall be official. However, ship(s) tank samples may also be tested for sediment and water, and for other quality aspects when required. The results of these ship(s) tank sample tests, together with the shore tank sample tests may be shown on the cargo certificate.
b) When discharging a ship, the pipeline sample taken from a properly designed and operated automatic line sampler in the discharge line shall be official. Where no proper line sample is available, the ship(s) tank sample will be official except where specifically exempted.

6.8 Finished Products

When loading or discharging finished products, take samples from both shipping and receiving tanks, and from the pipeline, if required.

6.8.1 *Ship or Barge Tanks*

Sample each product after the vessel is loaded or just before unloading.

6.8.2 *Tank Cars*

Sample the product after the car is loaded or just before unloading.

6.8.3 *Package Lots (Cans, Drums, Barrels, or Boxes)*

Take samples from a sufficient number of the individual packages to prepare a composite sample that will be representative of the entire lot or shipment. Select at random the individual packages to be sampled. The number of such random packages will depend upon several practical considerations, such as,

a) The stringency of the product specifications;
b) The source and type of the material and whether or not more than one production batch may be represented in the lot;
c) Previous experience with similar shipments, particularly with respect to the uniformity of quality from package to package; and
d) In most cases, the number specified in Table 2 will be satisfactory.

Table 2 Minimum Number of Packages to be Selected for Sapling

[*Clause* 6.8.3 (d)]

Sl No. (1)	Number of Packages in Lot (2)	Number of Packages to be Sampled (3)
i)	1 to 3	all
ii)	4 to 64	4
iii)	65 to 125	5
iv)	126 to 216	6
v)	217 to 343	7
vi)	344 to 512	8
vii)	513 to 729	9
viii)	730 to 1 000	10
ix)	1 001 to 1 331	11
x)	1 332 to 1 728	12
xi)	1 729 to 2 197	13
xii)	2 198 to 2 744	14
xiii)	2 745 to 3 375	15
xiv)	3 376 to 4 096	16
xv)	4 097 to 4 913	17
xvi)	4 914 to 5 832	18
xvii)	5 833 to 6 859	19
xviii)	6 860 or above	20

6.9 Obtaining Samples

6.9.1 Extreme care and good judgment are necessary to ensure samples are obtained that represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn but only when absolutely necessary, such as in cold weather, or when handling at a high temperature, or for reasons of safety. Select wiping clothes so that lint is not introduced, which would contaminate samples.

6.9.2 As many petroleum vapours are toxic and flammable, avoid breathing them or igniting them from an open flame or a spark produced by static. Follow all safety precautions specific to the material being sampled.

6.9.3 When sampling relatively volatile products of more than 13.8 kPa RVP, fill and allow the sampling apparatus to drain before drawing the sample. If the sample is to be transferred to another container, also rinse this container with some of the volatile product and then drain. When the actual sample is emptied in to this container, suspend the sampling apparatus into the opening of the sample container and allow to remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

6.9.4 When sampling non-volatile liquid products of 13.8 kPa RVP or less, the sampling apparatus shall be filled and allowed to drain before drawing the actual sample. If the actual sample is to be transferred to another container, rinse the sample container with some of the product to be sampled and drain before it is filled with the actual sample.

> NOTE — When taking samples from tanks suspected of containing flammable atmospheres, precautions should be taken to guard against ignitions due to static electricity. Metal or conductive objects such as gage tapes, sample containers, and thermometers, should not be lowered into or suspected in, a compartment or tank which is being filled or immediately after cessation of pumping. A waiting period will generally permit a substantial relaxation of the electrostatic charge.

6.10 Handling Samples

6.10.1 *Volatile Samples*

It is necessary to protect all volatile samples of petroleum and petroleum products from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when the material is being transferred. When samples of more than 210 kPa RVP are being obtained, be sure to use containers strong enough to meet local safety regulations. After delivery to the laboratory, volatile samples should be cooled before the container is opened.

6.10.2 *Light Sensitive Samples*

It is important that samples sensitive to light, such as gasoline containing tetraethyl lead, be kept in the dark, if the testing is to include the determination of such properties as colour, tetraethyl lead and inhibitor contents, sludge-forming characteristics, stability tests, or neutralization value. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately. It is a definite advantage to use covered cardboard cartons into which the sample bottles may be placed immediately after collection.

6.10.3 *Refined Materials*

Protect highly refined products from moisture and dust by placing paper, plastic, or metal foil over the stopper and the top of the container.

6.10.4 *Container Outage*

Never completely fill a sample container, but allow adequate room for expansion, taking into consideration the temperature of the liquid at the time of filling and the probable maximum temperature to which the filled container may be subjected. Adequate sample mixing is difficult if the container is more than 80 percent full.

6.11 Shipping Samples

To prevent loss of liquid and vapour during shipment, and to protect against moisture and dust, cover the stoppers of glass bottles with plastic caps that have been swelled in water, wipe dry, place over the tops of the stoppered bottles, and allow to shrink tightly in place. Before filling metal containers, inspect the lip and caps for dents, out-of-roundness or other imperfections. Screw the caps of metal containers down tightly and check for leakage. Appropriate regulations applying to the shipment of flammable liquids must be observed.

6.12 Labelling Sample Containers

Label the container immediately after a sample is obtained. Use waterproof and oil proof ink or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing, and handling. Include the following information:

a) Date and time sample was taken (the period elapsed during continuous sampling and the hour and minute of collection for dipper samples);
b) Name of the individual who obtained the sample;
c) Tank/Vessel/Pipeline/container identifier or description;
d) Product name;
e) Reference symbol or identification number;
f) Type of sample; and
g) Quantity represented (tank /vessel).

7 SAMPLING PROCEDURE

7.1 The standard sampling procedures described in this method are summarized in Foreword. Alternative sampling procedures may be used if a mutually satisfactory agreement has been reached by the parties involved. Such agreement shall be put in writing and signed by authorized officials.

7.2 Bottle or Beaker Sampling

7.2.1 *Application*

The bottle or beaker sampling procedure is applicable for sampling liquids of 101 kPa RVP or less in tank cars, tank trucks, shore tanks, ship tanks, and barge tanks. Solids or semi-liquid that can be liquefied by heat may be sampled by this procedure, provided they are true liquids at the time of sampling.

7.2.2 *Apparatus*

A suitable sampling bottle or beaker, as shown in Fig. 2 is required. Recommended uses and diameter of openings in the bottle or beaker are given in Table 3.

Table 3 Weighted Sampling Bottle or Beaker

(*Clause* 7.2.2)

Sl No. (1)	Material (2)	Diameter of Opening mm (3)
i)	Light lubricating oils, kerosenes, gasolines, transparent gas oils, diesel fuels and distillates	20
ii)	Heavy lubricating oils, non-transparent gas oils	40
iii)	Light crude oils viscosity less than 43 mm^2/s at 40 °C	20
iv)	Heavy crude and fuel oils	40

7.2.3 *Procedure*

7.2.3.1 *All level sample (one-way)*

Inspect the sampling bottle or beaker, graduated cylinder and sample container for cleanliness and use only clean dry equipment. Obtain the estimate of the liquid level in the tank.

Lower the weighted, stoppered bottle, or beaker as near as possible to the draw-off level, pull out the stopper with a sharp jerk of the line and raise the bottle at a uniform rate so that it is about three-fourths full as it emerges from the liquid. For light products or deep tanks, a restricted opening may be needed to avoid filling the bottle.

7.2.3.2 *Running sample (two-way)*

Lower the unsupported bottle or beaker as near as possible to the level of the bottom of the outlet connection or swing line and then raise the bottle or beaker to the top of the oil at a uniform rate of speed such that it is about three-fourths full when withdrawn from the oil. For light products or deep tanks, a notched cork or other restricted opening may be needed to avoid filling the bottle.

7.2.3.3 *Top, upper, middle, lower and outlet samples*

Lower the weighted, stoppered bottle to the required top, upper, middle, lower, and outlet depths as shown in Fig. 1.

NOTE — Where emulsions are in relatively higher concentration in the bottom portions of the tank, the lower sample would not be considered representative of this lower third. Automatic line sampling is recommended in such cases. If this is not possible an outlet sample or bottom sample should be required instead of the lower sample. In addition, make a bottom thieving for both opening and closing gages so that any change in the S&W level at the bottom of the tank may be observed and noted.

a) At the selected level, pull out the stopper with a sharp jerk of the line and allow the bottle or beaker to fill, completely. When judged full, raise the bottle or beaker, pour off a small amount, and stopper immediately.

b) Where tank samples must be used for crude oil fiscalization and the tanks do not have swing suction lines or weirs, it is recommended that upper, middle, and outlet samples be taken. Test these samples and report separately. The S&W deduction should be the average of the three values. Also average other analyses.

7.2.3.4 *Multiple tank composite sample*

Prepare a composite sample in the laboratory (not in the field) by mixing portions of the all levels samples as specified in **3.1.18.**

7.2.3.5 *Composite spot sample*

Prepare a composite spot sample by mixing spot samples in equal proportions as specified in **3.1.19**, using either the three-way or the two-way procedure, whichever applies.

7.2.3.6 *Middle spot sample*

Obtain this sample in the manner specified in **3.1.20.**

7.2.3.7 *Handling*

Stopper and label bottle sample, immediately after taking them, and deliver to the laboratory in the original sampling bottles. There is an advantage to using a bottle and a sampling cage instead of a weighted beaker for volatile products. The loss of light ends is likely to occur when transferring the sample from a weighted beaker to another container.

7.3 Tap Sampling

7.3.1 *Application*

The tap sampling procedure is applicable for sampling liquids of 101 kPa RVP or less in tanks that are equipped with suitable, sampling taps or lines. This procedure is recommended for volatile stocks in tanks of the breather and balloon-roof type, spheroids, etc. (samples may be taken from the drain cocks of gage glasses, if the tank is not equipped with sampling taps). The assembly for tap sampling is shown in Fig. 3.

NOTE — If RVP is more than 110 kPa but not more than 179 kPa, a cooling bath as shown in Fig. 8 shall be used between the tank tap and the sample container to cool the sample and container and prevent volatilization of low-boiling components.

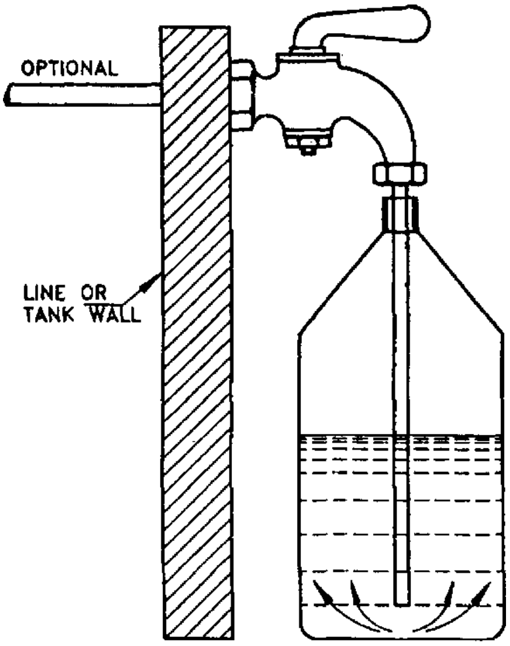


FIG. 3 ASSEMBLY FOR TAP SAMPLING

7.3.2 *Apparatus*

7.3.2.1 *Tank taps*

The tank should be equipped with at least three sampling taps placed equidistant throughout the tank height. On tanks that are not equipped with floating roofs each sample tap should extend into the tank a minimum of 10 cm. A sample tap should be equipped with a delivery tube which permit the filling of the sample container from the bottom.

7.3.2.2 *Sample cocks*

Samples of crude petroleum may be taken through sample cocks properly placed in the shell of the tank. The upper sample cock shall be located 457 mm below the top of the tank shell; the lower sample cock shall be located level with the bottom of the outlet elbow or other similar fitting if installed on the outlet connection; and the middle sample cock shall be located halfway between the upper and lower sample cocks. An additional cock for the clearance sample shall be located 102 mm below the bottom of the Outlet connection to determine whether the level of merchantable oil is at least below this point. The sample cocks shall be located a minimum of 1.8 m distant circumferential from the pipeline outlet and drain 2.4 m from the filling line connection. The sample, cocks shall be of 19.5 mm size, and the lines shall be of 19.05 mm nominal diameter for crude oil of 18° API gravity or less. For lighter oil, 12.7 mm size cocks, with 12.7 mm nominal diameter lines, shall be used. The lines shall extend a minimum of 102 mm inside the tank shell, except on floating-roof tanks, where flush installations are necessary. All sample cocks shall be equipped with sealable valves and plugged inspection tees.

On tanks of more than 1 590 m^3 (10 000 barrel) capacity, at least two sets of sample cocks shall be installed, located equidistant around the circumference of the tank. Five or more sample cocks should be installed per set, evenly spaced between lower and upper sample levels.

7.3.2.3 *Tube*

A delivery tube that will not contaminate the product being sampled and long enough to reach to the bottom of the sample container is required to allow submerged filling. When a cooling bath is used while tap sampling, a similar suitable tube should be used between the tank tap and the cooler inlet.

7.3.2.4 *Sample containers*

Use clean, dry glass bottles of convenient size and strength to receive the samples. If the vapour pressure of the product to be sampled is between 110 and 179 kPa RVP, protect the bottle with a metal cover until the sample is discarded. In some cases, such as the sampling of crude petroleum, metal containers may be used instead of glass bottles.

7.3.3 *Procedure*

7.3.3.1 Before a sample is drawn, flush the tap (or gage glass drain cock) and line until they are purged completely. Connect the clean delivery tube to the tap. Draw upper, middle, or lower samples directly from the respective taps after the flushing operation. Stopper and label the sample container immediately after filling, and deliver it to the laboratory.

7.3.3.2 When a sample cooler is used during the tap sampling operation, flush the tap (or gage glass drain cock). Then, using a section of clean tubing, connect the tap to the cooler inlet. Flush the cooler thoroughly, after which connect the clean delivery tube to the cooler outlet and proceed with the sampling operation.

7.3.3.3 In the sampling of crude petroleum, check for merchantable oil at the clearance sample cock. Flush each sample connection until all oil from the previous run has been removed and the sample lines are filled with fresh oil from the tank.

a) On tanks of 1 590 m^3 (10 000 barrel) capacity or smaller, take samples of equal amounts from the lower, middle and upper sample connections. A measuring cup of proper size may be used to assure the drawing of the proper quantity from each sample cock.

b) On tanks of more than 1 590 m^3 (10 000 barrel) capacity, take samples of equal amounts from each of the sample connections at each set of sample connections.

c) Mix all samples in equal proportions for a composite sample, or test the samples separately and average the results.

d) When crude oil in a tank fails to reach the upper or middle sample cocks on a tank equipped with three sample cocks, it is suggested that the sample for the run be obtained as follows:

    1) If the level of the oil is nearer the upper sample cock than the middle, take two-thirds of the example from the middle sample cock and one third from the lower;

    2) If the level of oil is nearer the middle sample cock than the upper, take one half of the sample from the middle and one half from the lower; and

    3) If the level of the oil is below the middle sample cock, take all the sample from the lower cock.

7.4 Manual Sampling of Pipeline Streams

7.4.1 *Application*

This manual line sampling procedure is applicable for sampling liquids of 101 kPa RVP or less and semi-liquids in pipelines, filling lines, and transfer lines. Where custody transfer is involved, continual automatic sampling is the preferred method. In the event of automatic sampler failure, manual samples may be needed. Take such manual samples as representatively as possible.

7.4.2 *Apparatus*

7.4.2.1 *Sampling probe*

The function of the sampling probe is to withdraw from the flowing stream a portion that will be representative of the entire stream. Probe designs that are commonly used are shown in Fig. 4. These are:

a) A tube extending to the centre of the line and bevelled at a 45° angle facing upstream (*see* Fig. 4A).

b) A short-radius forged elbow or pipes bend extending to the centre line of the pipe and facing upstream. The end of the probe should he reamed with to give a sharp entrance edge (*see* Fig. 4B); and

c) A closed-end tube with a round orifice spaced near the closed end that should be positioned in such a way that the orifice is in the centre of the pipeline and is facing the stream as shown in Fig. 4C.

7.4.3 *Probe Location*

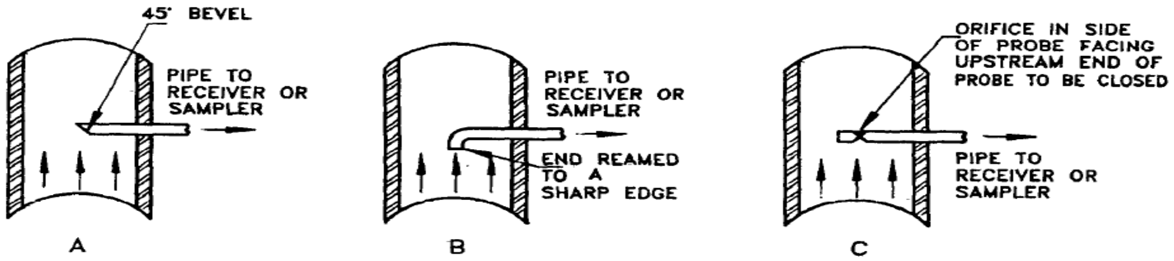
Since the fluid to be sampled may not always be homogeneous, the location, the position and the size of the sampling probe should be such as to minimize any separation of water and heavier particles that would make their concentration different in the gathered sample than in the main stream.

7.4.3.1 The probe should always be in a horizontal position to prevent drain back of any part of the sample to the main stream.

7.4.3.2 The sampling probe should be located preferably in a vertical run of pipe where such a vertical run can be provided. The probe may also be located in a horizontal run of pipe provided the flowing velocity is high enough to provide adequate turbulent mixing. While adequate flowing velocity may not eliminate a concentration difference between the bottom of the pipe and the top of the pipe, it may provide an average concentration at the mid-pipe probe location that will be representative of the entire stream at the sampling station.

7.4.3.3 Where adequate flowing velocity is not available, a suitable device for mixing the fluid flow should be installed upstream of the sampling tap to reduce stratification to an acceptable level. If flow has been vertical for a sufficient distance as in a platform riser, such a device may not be necessary even at low-flow rates. Some effective devices for obtaining adequate mixing area reduction in pipe size; a series of baffles, an orifice or perforated plate, or a combination of any of these methods. The design or sizing of the device is optional with the user, as long as the flowing stream is sufficiently well mixed to provide a representative sample from the probe.

7.4.3.4 The sampling point should be as near as practicable to the point where the oil passes to the sample receiver.


45° BEVEL
PIPE TO RECEIVER OR SAMPLER
A
PIPE TO RECEIVER OR SAMPLER
END REAMED TO A SHARP EDGE
B
ORIFICE IN SIDE OF PROBE FACING UPSTREAM END OF PROBE TO BE CLOSED
PIPE TO RECEIVER OR SAMPLER
C

NOTE — Probes may be fitted with valves or plug cocks. The probe should be oriented horizontally.

FIG. 4 PROBES FOR CONTINUOUS SAMPLING

7.4.3.5 The sampling lines should be as short as practicable and should be cleared before any samples are taken.

7.4.3.6 To control the rate at which the sample is withdrawn, the probe should be fitted with valves or plug.

7.4.4 *Procedure*

7.4.4.1 Adjust the valve or plug cock from the sampling probe, so that a steady stream is drawn from the probe. Whenever possible, the rate of sample, withdrawal should be such that the velocity of liquid flowing through the probe is approximately equal to the average linear velocity the stream flowing through the pipeline. Measure and record the rate of sample withdrawal as litres per hour. Divert the sample stream to the sampling container continuously or intermittently to provide a quantity of sample that will be of sufficient size for analysis.

7.4.4.2 In sampling crude petroleum, samples of 0.25 litre or more should be taken every hour or less, whichever is thought necessary. By mutual agreement, the sample period or sample size, or both, may be varied to accommodate the parcel size. It is important that the size of the samples and the intervals between the sampling operations be uniform for a uniform-flow rate. When the mainstream flow rate is variable, vary the sampling rate accordingly so that the amount of sample is always withdrawn from any given amount of fluid passing the sampling point in the mainstream. In practice, this is difficult to accomplish.

a) Place the sample of crude petroleum in a closed container and at the end of the agreed upon time period, mix the combined sample and take a composite sample for test purposes. Refer to **6.6** for mixing and handling. Store the sample container in a cool, dry place; avoid exposure to direct sunlight.
b) Alternatively, line sample may be taken at regular intervals and individually tested. The individual test results may be arithmetically averaged, adjusting for variations in flow rate during the agreed upon time period.
c) Either composited or arithmetically averaged results arc acceptable.

7.4.4.3 With either procedure, always label the sample and deliver it to the laboratory in the container in which it was collected.

NOTE — When sampling semi-liquids, heat the sampler lines, valves, and receiver to a temperature just sufficient to keep the material, liquid and to assure accurate sampling and mixing.

7.5 Dipper Sampling

7.5.1 *Application*

The dipper sampling procedure is applicable for sampling liquids of 13.8 kPa RVP or less and semi-liquids where a free or open-discharge stream exists, as in small filling and transfer pipelines (500 mm diameter or less) and filling apparatus for barrels, packages and cans.

7.5.2 *Apparatus*

7.5.2.1 *Dipper*

Use a dipper with a flared bowl and a handle of convenient length, made of material such as tinned steel that will not affect the product being tested. The dipper should have a capacity suitable for the amount to be collected and must be protected from dust and dirt when not being used.

7.5.2.2 *Sample container*

Use a clean, dry container of the desired size.

7.5.3 *Procedure*

Insert the dipper in the free-flowing stream so that a portion is collected from the full cross-section of the stream. Take portions at time intervals chosen so that a complete sample proportional to the pumped quantity is collected. The gross amount of sample collected should be approximately 0.1 percent but not more than 150 litre, of the total quantity being sampled. Transfer the portions into the sample container as soon as collected. Keep the container closed, except when pouring a dipper portion into it. As soon as all portions of the sample have been collected, close and label the sample, container, and deliver it to the laboratory.

7.6 Tube Sampling

7.6.1 *Application*

The tube sampling procedure is applicable for sampling liquids of 13.8 kPa RVP or less and semi-liquids in drums, barrels and cans.

7.6.2 *Apparatus*

7.6.2.1 *Tube*

Either a glass or metal tube may be used, designed so that it will reach to within about 3 mm of the bottom and have a capacity of approximately 0.5 or 1.0 litre. A metal tube suitable for sampling 190 litre drums is shown in Fig. 5. Two rings soldered to opposite sides of the tube at the upper end are convenient for holding it by slipping two fingers through the rings, thus leaving the thumb free to close the opening.

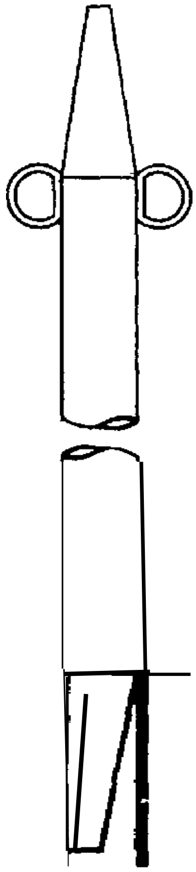


FIG. 5 SAMPLING TUBE

7.6.3 *Sample Containers*

Use clean, dry cans or glass bottles.

7.6.3.1 *Drums and barrels*

Place the drum or barrel on its side with the bung up. If the drum does not have a side bung, stand it upright and sample from the top. If detection of water, rust, or other insoluble contaminants is desired, let the barrel or drum remain in this position long enough to permit the contaminants to settle. Remove the bung and place it beside the bung hole with the oily side up. Close the upper end of the clean, dry sampling tube with the thumb and lower the tube into the oil for a depth of about 0.3 m. Remove the thumb, allowing oil to flow into the tube. Again close the upper end with the thumb and withdraw the tube. Rinse, the tube with the oil by holding it nearly horizontal and turning it so that the oil comes in contact with that part of the inside surface that will be immersed when the sample is taken. Avoid handling any part of the tube that will be immersed in the oil during the sampling operation. Discard the rinse oil and allow the tube to drain. Insert the tube into the oil again, holding the thumb against the upper end (if an all levels sample is desired, insert the tube with the upper end open). When the tube reaches the bottom, remove the thumb and allow the tube to fill. Replace the thumb, withdraw the tube quickly, and transfer the contents to the sample container. Do not allow the hands to come in contact with any part of the sample. Close the sample container, replace and tighten the bung in the drum or barrel. Label the sample container and deliver it to the laboratory.

7.6.3.2 *Cans*

Obtain samples from cans of 19 litre capacity or larger in the same manner as from drums and barrels using a tube of proportionately smaller dimensions. For cans of less than 19 litre capacity, use the entire contents as the sample, selecting cans at random as indicated in Table 2 or in accordance with agreement between the purchaser and the seller.

7.7 Thief Sampling

7.7.1 *Application*

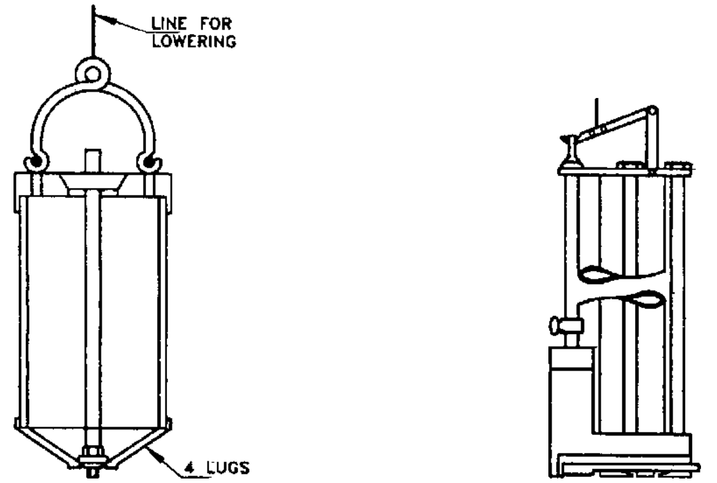
The thief sampling procedure is applicable for obtaining bottom samples (*see* Fig. 1) or of semi-liquids in tank cars and storage tanks. The thief is also widely used in sampling crude petroleum in storage tanks. In this application it may be used for taking samples at different levels as well as for bottom samples of non-merchantable oil and water at the bottom of the tank.

7.7.2 *Apparatus*

7.7.2.1 *Thief*

The thief shall be designed so that a sample can be obtained within 13 mm of the bottom of the car or tank. Two types of thiefs are illustrated in Fig. 6. One type is lowered into the tank with valves open to permit the oil to flush through the container. When the thief strikes the bottom of the tank, the valves shut automatically to trap a bottom sample. The other type has a projecting stem on the valve rod which open the valves automatically as the stem strikes the bottom of the tank. The sample enters the container through the bottom valve and air is released simultaneously through the top. The valves snap shut when the thief is withdrawn. A core type thief similar to that shown in Fig. 6B with a uniform cross-section and bottom closure and with a capacity depending upon the size of the sample required, may be used for sampling crude petroleum. The thief should be capable of penetrating the oil in the tank to the required level, mechanically equipped to permit filling at any desired level, and capable of being withdrawn without undue contamination of the content. The thief may be equipped with the following:

a) Sample cocks for obtaining samples for the determination of water and sediment.



A
Bomb-Type Sampling Thief

B
Core-Type Sampling Thief

FIG. 6 SAMPLING THIEFS

b) Extension rods for use in obtaining samples at levels corresponding with requirements for high connections or for samples to determine high settled water and sediment levels.
c) Water and sediment gage for determining the height of water and sediment in the thief.
d) Windshield to be used when taking the gravity and temperature of the oil.
e) Opener to break the tension on the valve or slide at any desired level.
f) A thief cord marked so that sample can be taken at any depth in the vertical cross-section of the tank.
g) Hook to hang the thief in the hatch vertically.

7.7.2.2 *Sample containers*

Use clean, dry cans or glass bottles.

7.7.3 *Procedure*

7.7.3.1 Lower the clean, dry thief through the dome of the tank car or tank hatch until it strikes the bottom. When full, remove the thief and transfer the contents to the sample container. Close and label the container immediately and deliver it to the laboratory.

7.7.3.2 In the sampling of crude petroleum lower the clean, dry thief slowly into the oil to the desired depth, trip the thief to secure the sample and rise slowly to avoid agitation. The proper depths for sampling are described in **4.1**.

7.8 Extended-Tube Sampling

7.8.1 *Application*

The extended-tube sampling procedure may be used only for obtaining bottom water samples.

NOTE — The user should be aware that the procedure is used for sampling bottom water primarily on ships and barges. The procedure may be used for sampling bottom water in shore tanks, but no specific guidelines for such use are available.

7.8.2 *Apparatus*

7.8.2.1 A typical extended-tube sampling assembly is shown in Fig. 7. The extended-tube sampler consists of a flexible tube connected to the suction of a manually operated pump. For support purposes and to establish a known sampling point, the tubing is attached to the weighted end of a conductive wire or tape such that the open end of the tube is located approximately 125 mm above the tip of the weight. The tubing and wire (or tape) shall be long enough to extend to the bottom (reference height) of the vessel or storage tank from which the sample is to be obtained. A grounding cable shall be provided for the assembly.

7.8.2.2 In addition to the sampler, a clean, dry bottle or other appropriate container is required to collect each sample.

7.8.3 *Procedure*

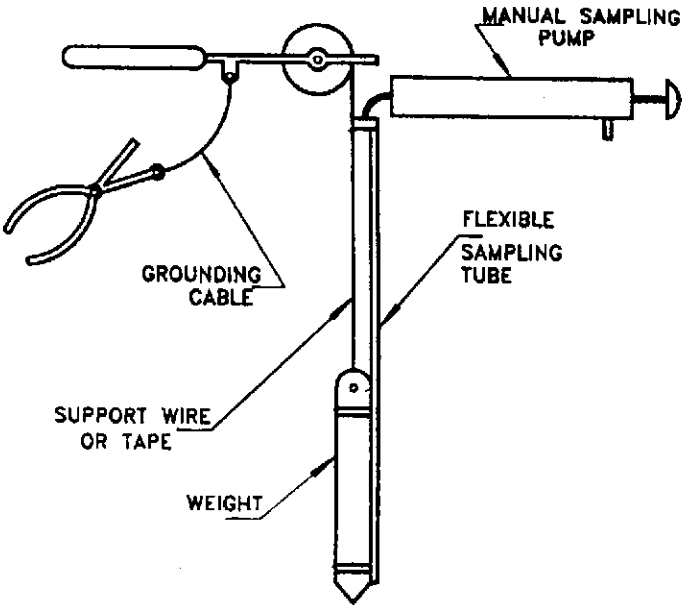


FIG. 7 TYPICAL EXTENDED-TUBE SAMPLER

7.8.3.1 *Assemble the extended-tube sampler*

7.8.3.2 Lower the weighted end of the sampling tube into a bucket of water and prime the sampler by operating the pump. When the assembly is satisfactorily primed, close-off (ensure it's not vented to the atmosphere), the top end of the assembly to prevent the loss of priming water as the sampling tube is removed from the bucket. Remove the sampling tube from the bucket, connect the grounding cable, to the ship or barge tank, and lower the weighted sampling tube to the bottom of the tank.

7.8.3.3 Begin the sampling operation by slowly and steadily operating the manual pump. To reduce the possibility of capturing a contaminated sample, collect a volume greater than twice the sampling assembly's capacity in a graduated cylinder or other measuring device and discard this volume. Collect the sample(s) directly in a clean, dry bottle(s) or other appropriate container(s).

NOTE — The capacity of the sampler may be calculated from the sampler, dimensions or may-be determined by measuring the volume of liquid discharge from a completely full assembly.

7.8.3.4 If a sample at a different level within the bottom water layer is required, raise the weighted bob and tubing to the new level above the bottom. Purge the residual water from the assembly and collect the new sample(s) as instructed above.

7.8.3.5 After each sample has been collected, immediately close and label the bottle (or container) in preparation for delivery to the laboratory.

7.8.3.6 When the sampling operation is complete, clean and disassembles the sampler components.

7.9 Nozzle Sampling

Nozzle sample at Fuel Station/Retail Outlet/Petrol Pump, etc, shall be representing the constituents present in the underground storage tank connected with the dispensing unit. Use suitable sample container as specified in **5** for nozzle sampling.

8 SAMPLING INDUSTRIAL AROMATIC HYDROCARBONS

8.1 Application

For obtaining samples of industrial aromatic hydrocarbons (benzene, toluene, xylene and solvent naphthas), proceed in accordance with **5** to **7** particularly those pertaining to precautions, care and cleanliness.

9 SAMPLING FOR SPECIFIC TESTS

9.1 Special Precautions

Special sampling precautions and instructions are required for some test methods and specifications. Such instructions supplement the general procedure of this method and supersede them if there is a conflict.

9.2 Distillation of Natural Gasoline

When obtaining samples of natural gasoline that are to be tested using IS 1448 (Part 18), the bottle sampling procedure **7** is preferred with the exception that pre-cooled bottles and laboratory composition is required. Before obtaining the sample, pre-cool the bottle by immersing it in the product, allow it to fill, and discard the first filling. If the bottle procedure cannot be used, obtain the sample by the tap procedure and with the use of the cooling bath as described in **7.2.3**. Do not agitate the bottle while drawing the sample. After obtaining the sample, close the bottle immediately with a tight-fitting stopper and store it in an ice bath or refrigerator at a temperature of 0 to 4.5 °C.

9.3 Reid Vapour Pressure

9.3.1 When sampling products that are to be tested using IS 1448 (Part 39) observe the following precautions and instructions.

9.3.1.1 *Precautions*

Vapour pressures are extremely sensitive to evaporation losses and to slight changes in composition. When obtaining, storing, or handling samples, observe the necessary precautions to ensure samples representative of the product and satisfactory for Reid vapour pressure tests. Official samples should be taken by or under the immediate supervision of, a person of judgment, skill, and sampling experience. If sampling or sample requirements for other tests differ from those described in **9.3.1.2** to **9.3.1.9**, obtain a separate sample for the Reid vapour pressure test. Never prepare composite samples for this test. Make certain that containers which are to be shipped by common carrier conform to state, or local regulations. When flushing or purging lines or containers, observe the pertinent regulations and precautions against fire, explosion, and other hazards.

9.3.1.2 *Cooling bath*

A bath (*see* Fig. 8) of sufficient size to hold the sample container and a cooling coil of about 7.6 m of copper tubing 9.5 mm or less in outside diameter shall be required when using the procedure described in **9.3.1.7**. One end of the coil is provided with a connection for attaching it to the tank sampling tap or valve. The other end is fitted with a suitable valve (outlet) of good quality. A removable copper tube of 9.5 mm or less in outside diameter and of sufficient length to reach the bottom of the sample container shall be connected to the open end of the outlet valve.

9.3.1.3 *Sample containers*

Use containers of not less than 0.9 litre and not more than 7.6 litre capacity, of sufficient strength to withstand the pressures to which they may be subjected, and of a type that will permit replacement of the cap or stopper with suitable connections for transferring the sample to the gasoline chamber of the vapour pressure apparatus. Open type containers have a single opening which permits sampling by immersion. Closed type containers have two openings; one in each end (or the equivalent thereof), fitted with valves suitable for sampling by water displacement or by purging.

9.3.1.4 *Transfer connections*

The transfer connection for the open-type container consists of an air tube and a liquid delivery tube assembled in a cap or stopper. The air tube extends to the bottom of the container. One end of the liquid delivery tube is flush with the inside face of the cap or stopper, and the tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred to the chamber. The transfer connection for the closed type container consists of a single tube with a connection suitable for attaching it to one of the openings of the sample container. The tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred.

9.3.1.5 *Sampling open tanks*

Use clean containers of the open type when sampling open tanks and tank cars. An all level sample obtained by the bottle procedure **7.2.3** is recommended. Before taking the sample, flush the container by immersing it in the product to be sampled. Then obtain the sample immediately. Pour off enough so that the container will be 70 to 80 percent full and close it promptly. Label the container and deliver it to the laboratory.

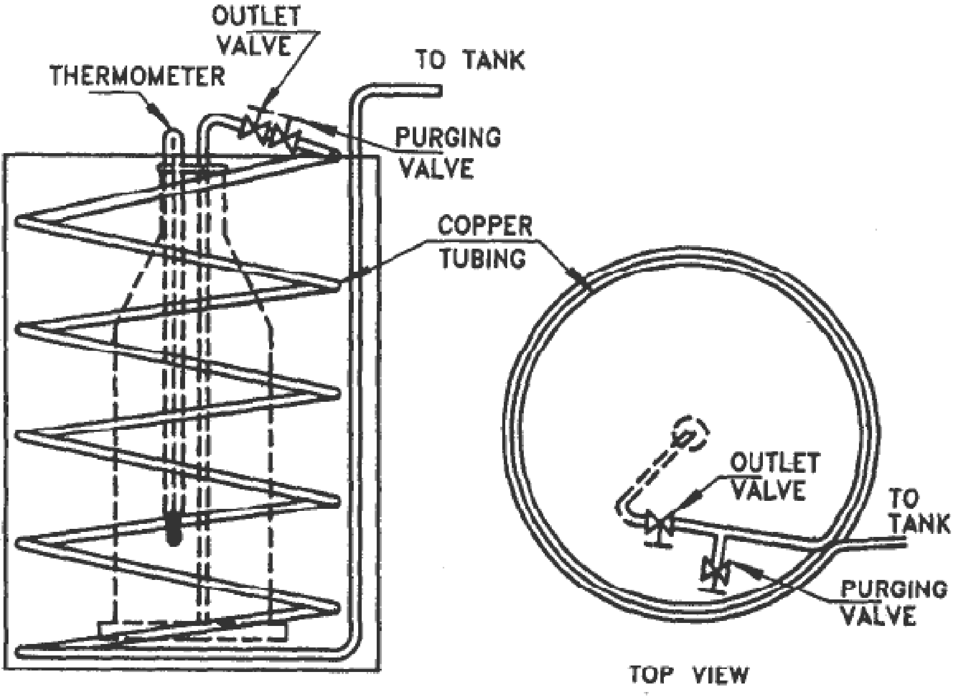


FIG. 8 COOLING BATH FOR REID VAPOUR PRESSURE SAMPLING

9.3.1.6 *Sampling closed tanks*

Containers of either the open or closed type may be used to obtain samples from closed or pressure tanks. If the open type is used, follow the cooling bath procedure described in **9.3.1.7**. If the closed type is used, obtain the sample using the water displacement procedure, **9.3.1.8** or the purging procedure, **9.3.1.9**. The water displacement procedure is preferable because the flow of product involved in the purging procedure may be hazardous.

9.3.1.7 *Cooling bath procedure*

When using a container of the open type, keep it at a temperature of 0 to 4.5 °C during the sampling operation by using the cooling bath (*see* Fig. 8). Connect the coil to the tank sampling tap or valve and flush it with a sufficient amount of product to ensure complete purging. When obtaining a sample, throttle the outlet valve so that the pressure in the coil will be approximately the same as that in the tank. Fill the container once to wash and cool it, and discard the wash product. Then draw the sample immediately. Pour off enough so that the container will be 70 to 80 percent full and close it promptly. Label the container and deliver it to the laboratory.

9.3.1.8 *Water displacement procedure*

Completely fill the closed-type container with water and close the valves. The water should be at the same temperature or lower than that of the product to be sampled. While permitting a small amount of product to flow through the fittings, connect the top or inlet valve of the container to the tank sampling tap or valve. Then open all valves on the inlet side of the container. Open the bottom or outlet valve slightly to allow the water to be displaced slowly by the sample entering the container. Regulate the flow so that there is no appreciable change in pressure within the container. Close the outlet valve as soon as gasoline discharges from the outlet; then in succession close the inlet valve and the sampling valve on the tank. Disconnect the container and withdraw enough of the contents so that it will be 70 to 80 percent full. If the vapour pressure of the product is not high enough to force liquid from the container, open both the upper and lower valves slightly to remove the excess. Promptly seal and label the container, and deliver it to the laboratory. The above is not applicable to LPG sampling.

9.3.1.9 *Purging procedure*

Connect the inlet valve of the closed-type container to the tank sampling tap or valve. Throttle the outlet valve of the container so that the pressure in it will be approximately equal to that in the container being sampled. Allow a volume of product equal to at least twice that of the container to flow through the sampling system. Then close all valves, the outlet valve first, the inlet valve of the container second, and the tank sampling valve last, and disconnect the container immediately. Withdraw enough of the contents so that the sample container will be 70 to 80 percent full. If the vapour pressure of the product is not high enough to force liquid from the container, open both the upper and lower valves slightly to remove the excess. Promptly seal and label the container and deliver it to the laboratory.

9.4 Oxidation Stability

9.4.1 When sampling products that are to be tested for oxidation stability, observe the following precautions and instructions.

9.4.1.1 *Precautions*

Very small amounts (as low as 0.001 percent) of some materials, such as inhibitors, have a considerable effect upon oxidation stability tests. Avoid contamination and exposure to light while taking and handling samples. To prevent undue agitation with air which promotes oxidation do not pour, shake or stir samples to any greater extent than necessary. Never expose them to temperatures above those necessitated by atmospheric conditions.

9.4.1.2 *Sample containers*

Use only brown glass or wrapped clear glass bottles as containers, since it is difficult to make certain that cans are free of contaminants, such as rust and soldering flux. Clean the bottles by the procedure described in **5.3** and if possible or required, further clean with cleaning mixture, and nearly saturate powdered potassium or sodium dichromate in concentrated sulphuric acid and allow it to stand for several hours, preferably 4 to 5 h. The acid is then poured off and thoroughly rinsed with distilled water; allow draining until dry and then protecting the bottles from dust and dirt.

9.4.1.3 *Sampling*

A running sample obtained by the bottle procedure prescribed in **7** is recommended because the sample is taken directly in the bottle. This reduces the possibility of air absorption, loss of vapours, and contamination. Just before sampling, rinse the bottle with the gasoline to be sampled.

9.5 Lacquer, Solvents and Diluents

9.5.1 When sampling bulk shipments of lacquer, solvents and diluents observe the following precautions and instructions.

9.5.1.1 *Tanks and tank cars*

Obtain upper and lower samples (*see* Fig. 1) of not more than 1.0 litre each by the bottle procedure prescribed in **7.2.3.2**. Prepare in the laboratory a composite sample of not less than 2.0 litre by mixing equal parts of the upper and lower samples.

9.5.1.2 *Barrels, drums, and cans*

Obtain samples from at least five percent of the number of containers in any shipment. The number of containers to be sampled may be increased at the discretion of the purchaser. In the case of expensive solvents which are purchased in small quantities, it is recommended that each container be sampled. Withdraw a portion from the centre of each container to be sampled with a clean tube (*see* **7.6.3**) or weighted bottle (*see* **7.2.3.2**) (A smaller bottle may be used). Prepare a composite sample of at least 1 litre by mixing equal portions of not less than 0.5 litre from each container sampled.

9.6 Asphaltic Materials

When sampling asphaltic materials obtain samples by the boring procedure or the grab procedure. A sample of sufficient size to yield at least 100 g of recovered bitumen is required. About 1 000 g of sheet asphalt mixtures usually will be sufficient. If the largest lumps in the sample are 25 mm, 2 000 g will usually be required and still larger samples if the mixtures contain larger aggregates.

9.7 Emulsified Asphalts

Obtain samples from tanks, tank cars, and tank trucks by the bottle sampling procedure prescribed in **6.2.3**, using a wide mouth (40 mm or greater) bottle. Use the dipper procedure prescribed in **6.5.3** to obtain samples from filling or discharge lines. Sample packages in accordance with Table 2. If the material is solid or semisolid, use the boring sampling procedure, as mentioned in IS 1447 (Part 3). Obtain at least 4 litre or 4.5 kg from each lot or shipment. Store the samples in clean, airtight containers at a temperature of not less than 4 °C until tested. Use glass or black iron containers for emulsified asphalts of the RS-1 type.

ANNEX A

(*Clauses* 1.3 *and* 6.1)

MANDATORY INFORMATION

A-1 PRECAUTIONARY STATEMENTS

A-1.1 The following substances may be used throughout the course of this standard test method. The precautionary statements should be read prior to use of such substances.

A-1.1.1 *Benzene*

Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Use fume hood whenever possible. Avoid build-up of vapours and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters. Avoid prolonged breathing of vapours or spray mist. Avoid contact with skin and eyes. Do not take internally.

A-1.1.2 *Diluents (Naphtha)*

Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid buildup of vapours and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters. Avoid prolonged breathing of vapours or spray mist. Avoid prolonged or repeated skin contact.

A-1.1.3 *Flammable Liquid (General)*

Keep away from heat, sparks and open flame. Keep container closed. Use only with adequate ventilation. Avoid prolonged breathing of vapour or spray mist. Avoid prolonged or repeated contact with skin.

A-1.1.4 *Gasoline (White)*

Harmful if absorbed through skin. Keep away from heat, sparks and open flame. Keep container closed. Use with adequate ventilation. Avoid build-up of vapours and eliminate all sources of ignition especially non-explosion proof electrical apparatus and heaters. Avoid prolonged breathing of vapour or spray mist. Avoid prolonged or repeated skin contact.

A-1.1.5 *Toluene and Xylene*

WARNING — Flammable. Vapour harmful.

Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid breathing of vapour or spray mist. Avoid prolonged or repeated contact with skin.

ANNEX B

(*Clauses* 5.1.2.4 *and* 6.5.3)

MANDATORY NFORMATION

SUMMARY OF CONTAINER RECOMMENDATION

Material/Type of Analysis	Thermal Stability[1)]	MSEP	Electrical Conductivity[1)]	Lubricity	Trace Metals	Copper Corrosion[1)]	Particulate
Hard borosilicate glass							
Immediate use	S	P	P	S	NR	S	NE
Storage	NE	P	P	S	NR	S	NE
Reuse	S	S	P	S	NR	S	NE
Soft soda lime glass							
Immediate use	NE	S	S	NE	NR	NE	NE
Storage	NR	NR	NR	NR	NR	NE	NE
Reuse	NR	S	S	NR	NR	NR	NE
Aluminium container							
Immediate use	NR	NR	NR	NR	NR	NR	NE
Storage	NR	NR	NR	NR	NR	NR	NE
Reuse	NR	NR	NR	NR	NR	NR	NE
Epoxy lined steel							
Immediate use	P	P	P	P	NR	S	P
Storage	P	P	P	P	NR	S	P
Reuse	P	P	P	P	NR	S	P
Polytetrafluoroethylene							
Immediate use	NE	S	NR	NR	P	S	NE
Storage	NE	NE	NR	NR	P	S	NE
Reuse	NE	NE	NR	NR	P	S	NE
Tin plate soldered steel							
Immediate use	S	S	S	S	NR	NR	NE
Storage	NR	NR	NR	NR	NR	NR	NE
Reuse	NR	NR	NR	NR	NR	NR	NE
High-density linear Polyethylene							
Immediate use	NR	S	NR	NR	P	S	NE
Storage	NR	NR	NR	NR	P	S	NE
Reuse	NR	NR	NR	NR	P	S	NE

[1)] All transparent or translucent containers must be shielded from light by wrapping with opaque material. P = preferred, S = suitable, NR = not recommended, NE = not evaluated but may be suitable.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehradun	Dr Anjan Ray (***Chairman***)
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Organization	*Representative(s)*
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Member Secretary

Shrimati Nagamani T.
Scientist 'E' (PCD), BIS

(*Continued from second cover*)

Each procedure is suitable for sampling a number of specific materials under definite storage, transportation, or container conditions. The basic principle of each procedure is to obtain a sample or a composite of several samples in such manner and from such locations in the tank or other container that the sample or composite will be truly representative of the petroleum or petroleum product.

IS 1447 'Methods of sampling of petroleum and its products' was initially published in 1966, based on the overseas publications IP Part IV 1962 Ed. 2 'Methods of sampling' issued by 'The Institute of Petroleum, London, ASTM D 270 'Sampling and petroleum products' and ASTM D 1265 'Sampling of liquefied petroleum (LP) Gases' issued by the American Society for Testing and Materials, respectively. Later with the advent of techniques, the Committee decided to revise the standard dividing it into four parts revision. Accordingly, this standard was revised for the first time, as IS 1447 (Part 1) in 2000. The other parts in this series are as follows:

(Part 2) : 2013	Liquefied petroleum gases - Method of sampling (LPG) (*second revision*)
(Part 3) : 1992	Method of sampling of semi-solid and solid petroleum products (*first revision*)
(Part 4) : 1989	Sampling of petroleum coke for laboratory analysis (*first revision*)

This second revision was taken up as a result of the review of the standard by the concerned Technical Committee. The sample containers and design of sample containers for jet fuel were included in this revision. Nozzle sampling procedure has also been included.

In the formulation of this standard, considerable assistance has been obtained from the joint publication ASTM D 4057 'Standard practice for manual sampling of petroleum and petroleum products', ISO 3170 'Petroleum liquids-Manual sampling', ASTM D 4306 'Aviation Fuel Sample Containers for Tests Affected by Trace Contamination' and API Designation: MPMS (Chapter 8.1) issued by the American Society for Testing and Materials (USA) and Petroleum Measurements Tables.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 01 (12514).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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